

---

# Demonstration of a High-Brightness Water-Window Laser-Plasma Source for Soft X-Ray Microscopy



Dale Martz  
Biomedical & X-Ray Physics  
Dept. of Applied Physics  
Royal Inst. of Technol. (KTH), Stockholm

---

# Demonstration of a high-brightness water-window laser-plasma source for soft x-ray microscopy

D. H. Martz<sup>1</sup>, O. von Hofsten<sup>1</sup>, M. Selin<sup>1</sup>, H. Legall<sup>2</sup>, G. Blobel<sup>2</sup>, C. Seim<sup>3</sup>, M. Bertilson<sup>1</sup>, H. Stiel<sup>2</sup>, U. Vogt<sup>1</sup>, and H. M. Hertz<sup>1</sup>

<sup>1</sup>Biomedical and X-Ray Physics, Dept. of Applied Physics, KTH Royal Inst. of Technology/Albanova, 10691 Stockholm, Sweden

<sup>2</sup>Max-Born-Institut, Max-Born-Straße 2A, 12489 Berlin, Germany

<sup>3</sup>Institute of Optics and Atomic Physics - Analytical X-ray physics, Technical University-Berlin, 10623 Berlin Germany

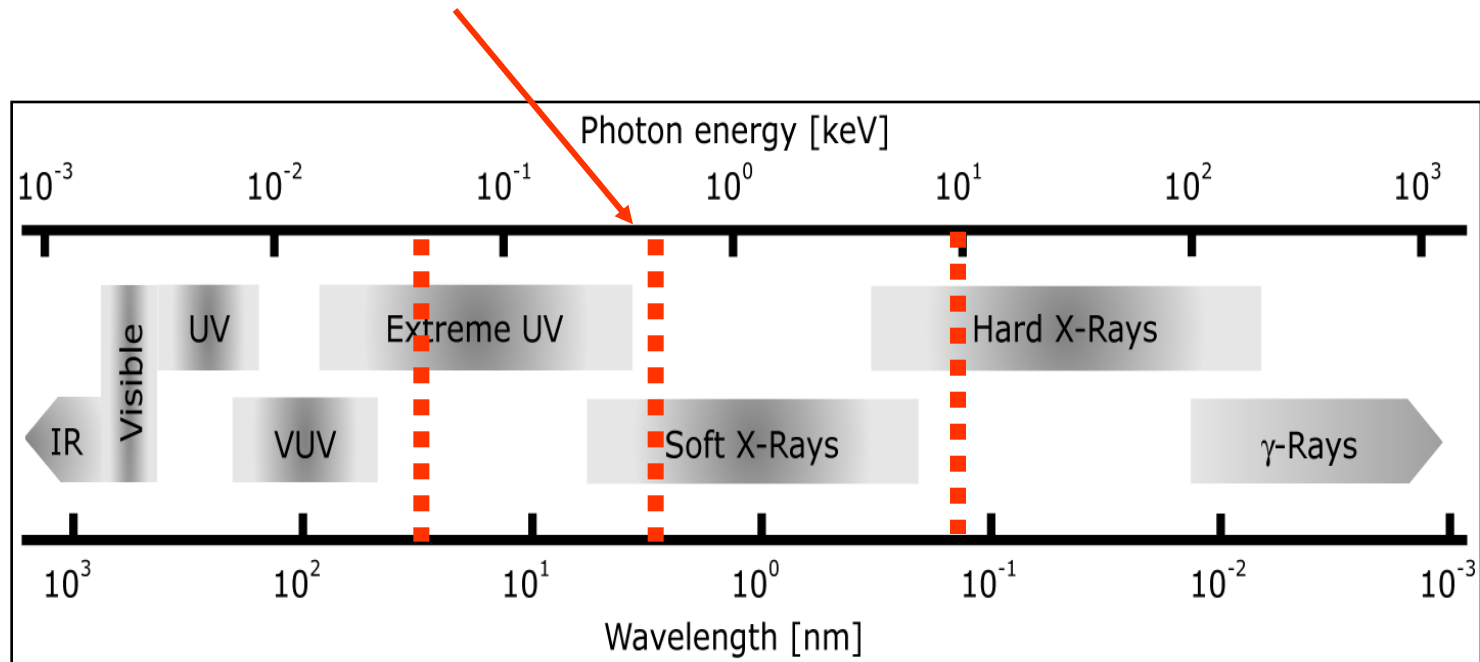
# Today

## Soft x-rays :

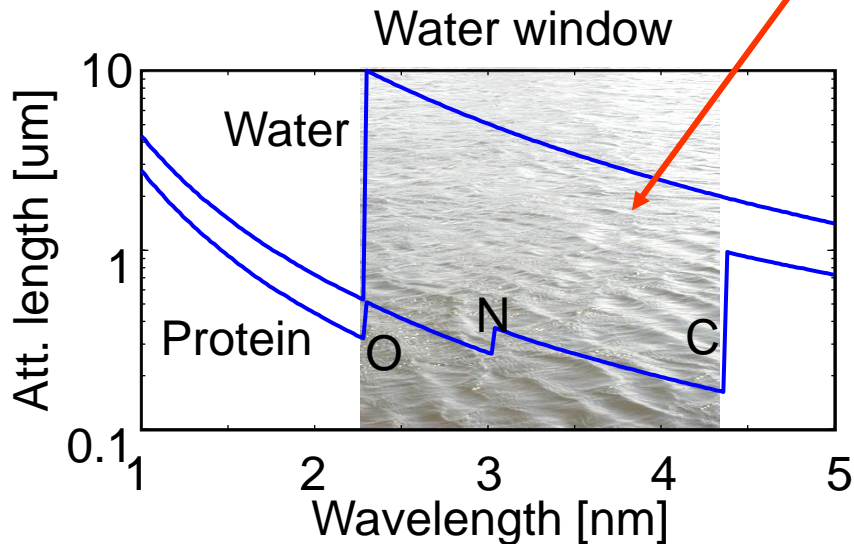
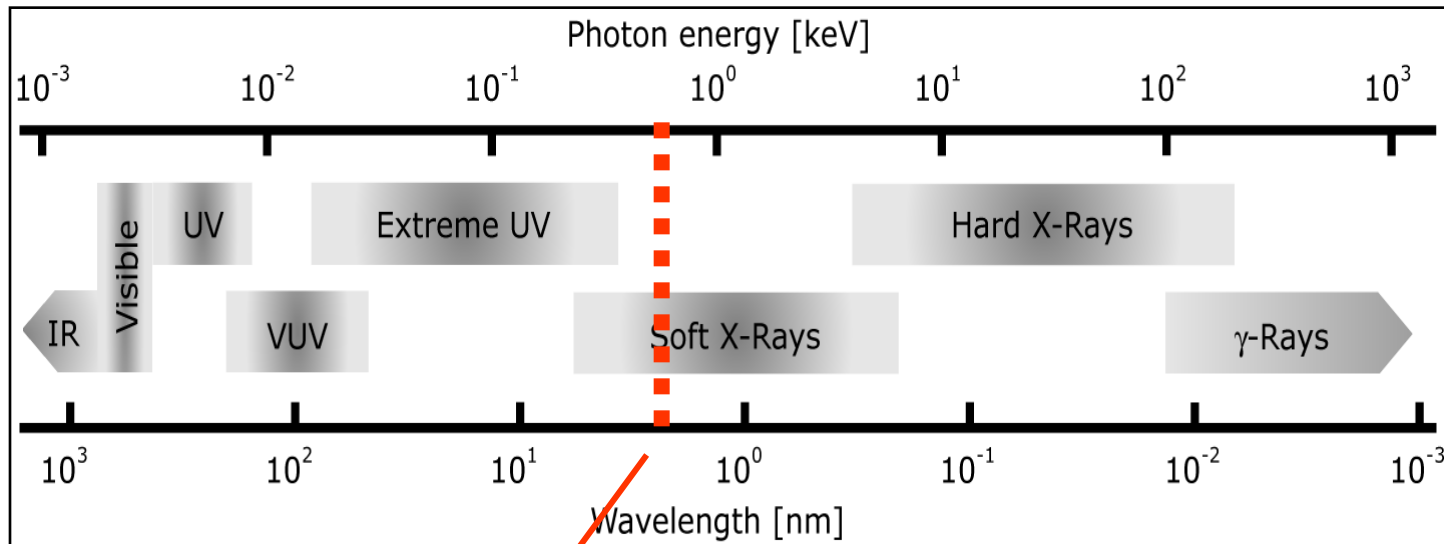
Liquid-jet laser-plasma sources

x-ray optics

Laboratory x-ray microscopy



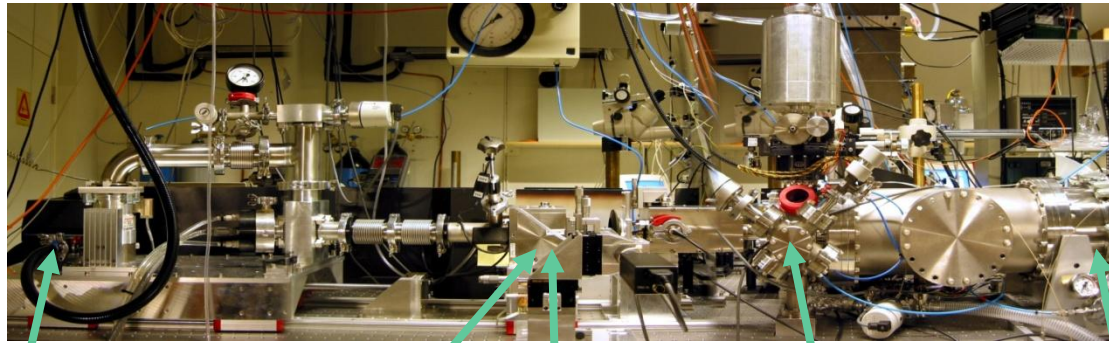
# Soft x-rays: Water-window x-ray microscopy



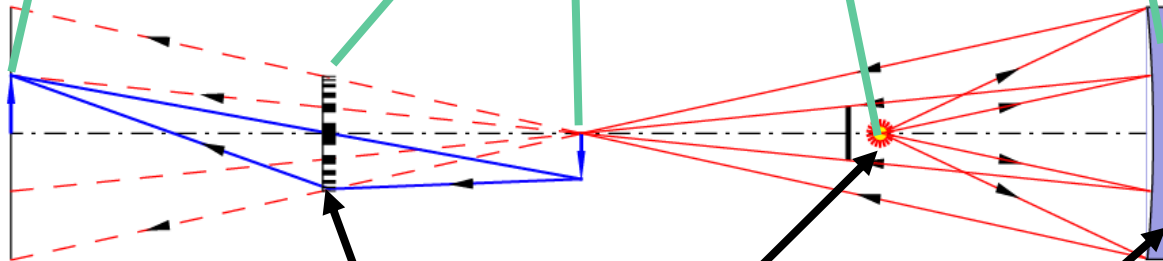
- + Resolution:  $0.61\lambda/NA$
- + Natural contrast for wet/frozen specimen
- + Possibility to study thick ( $\sim 10 \mu\text{m}$ ) objects
- Lack of laboratory high-brightness sources
- Inefficient optics

Soft x-rays:

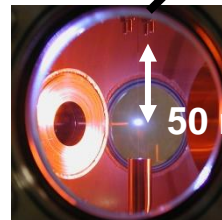
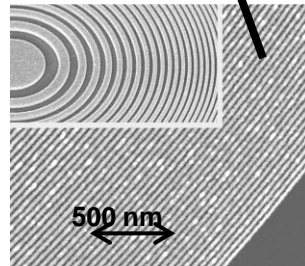
# Laboratory water-window x-ray microscopy



$\lambda=3.37$  nm &  
 $\lambda=2.48$  nm



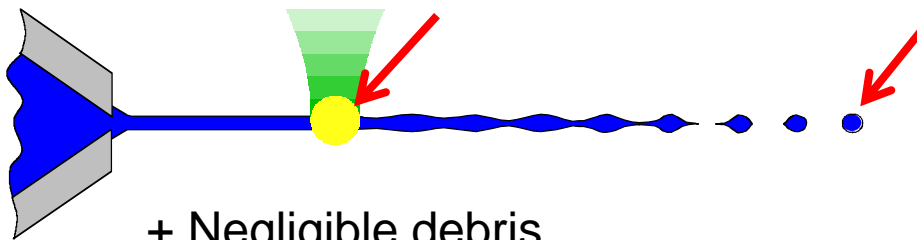
Micro zone plates for  
high-resolution imaging



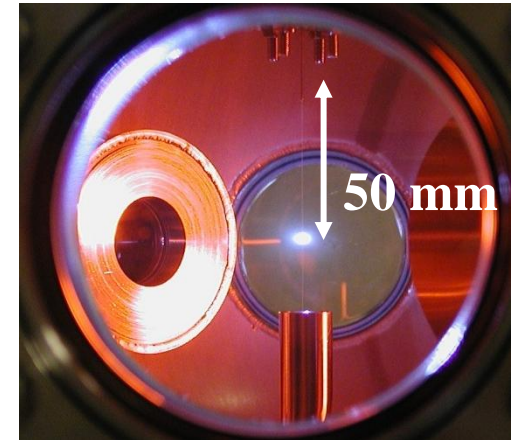
Normal-incidence  
multilayer mirrors  
as condensers

Berglund et al, J. Microsc. (2000), Johansson et al, RSI (2002) Takman et al, J. Microsc. (2007)

# Laboratory soft x-ray sources: Liquid-jet/droplet laser plasmas

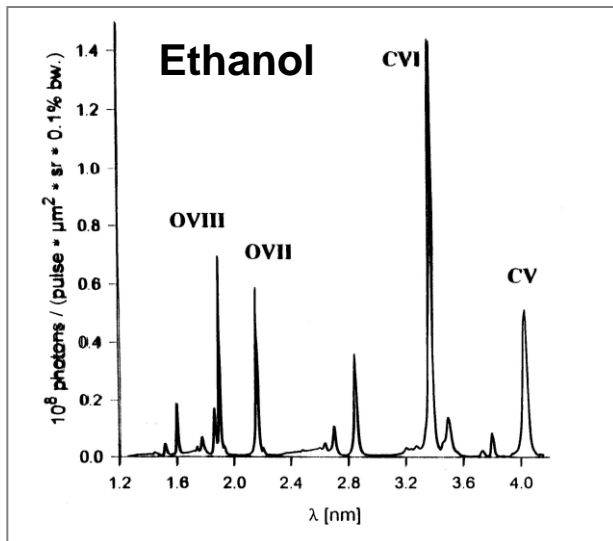


- + Negligible debris
- + Regenerative target
- + High rep.-rate operation
- + High-power operation possible
- + Tailored spectral emission



Rymell et al, Opt. Commun. (1993); Malmqvist et al, RSI (1996)

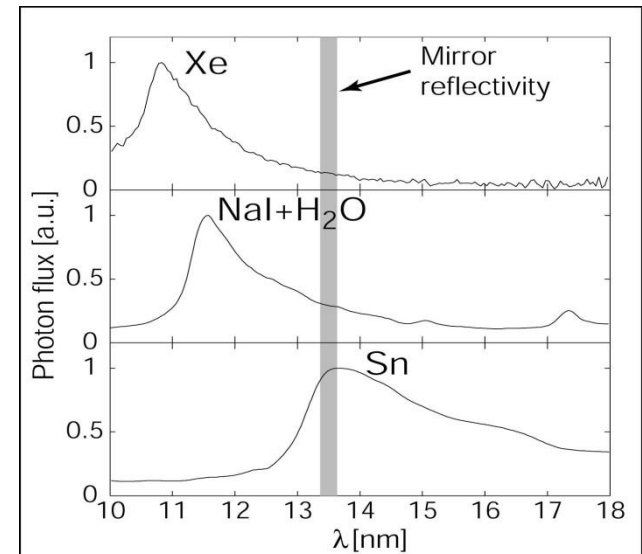
## $\lambda \approx 2-4$ nm: Water-window



- + Line emission  $\Rightarrow$  fixed wavelength
- +  $\lambda/\Delta\lambda > 500$
- + High brightness

Rymell et al, APL (1995), Berglund et al APL (1997)

## $\lambda = 13$ nm: EUV Lithography



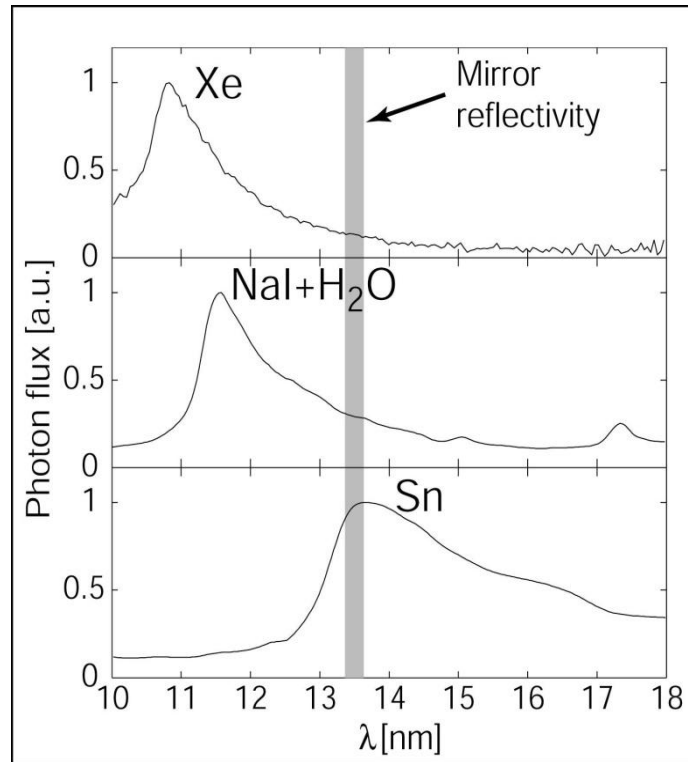
Hansson et al, MNE (2001); Jansson et al APL (2004)

# EUV sources: Liquid tin jet

## Stable jet @ >250 C



## Spectral match



CE: 2.5% into (2%BW $\times$ 2 $\pi$  $\times$ sr)

## Debris:



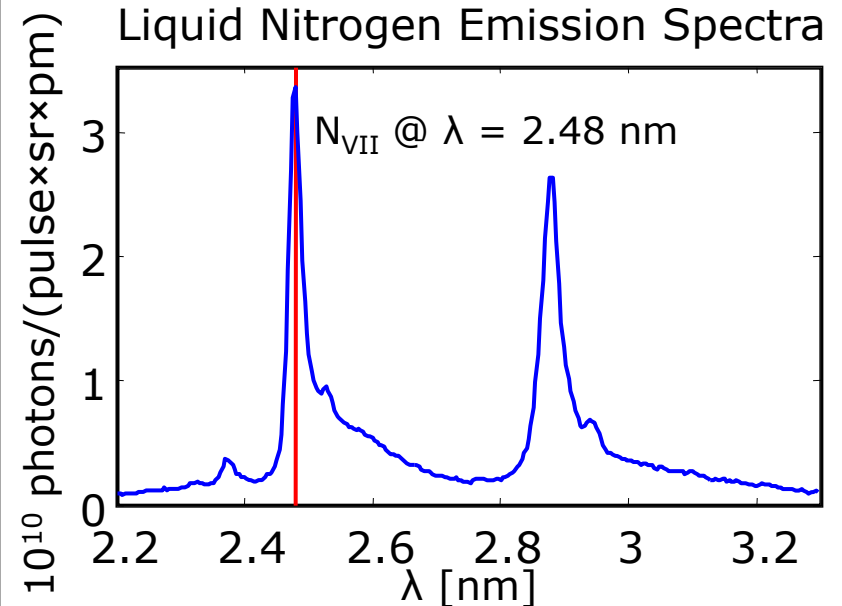
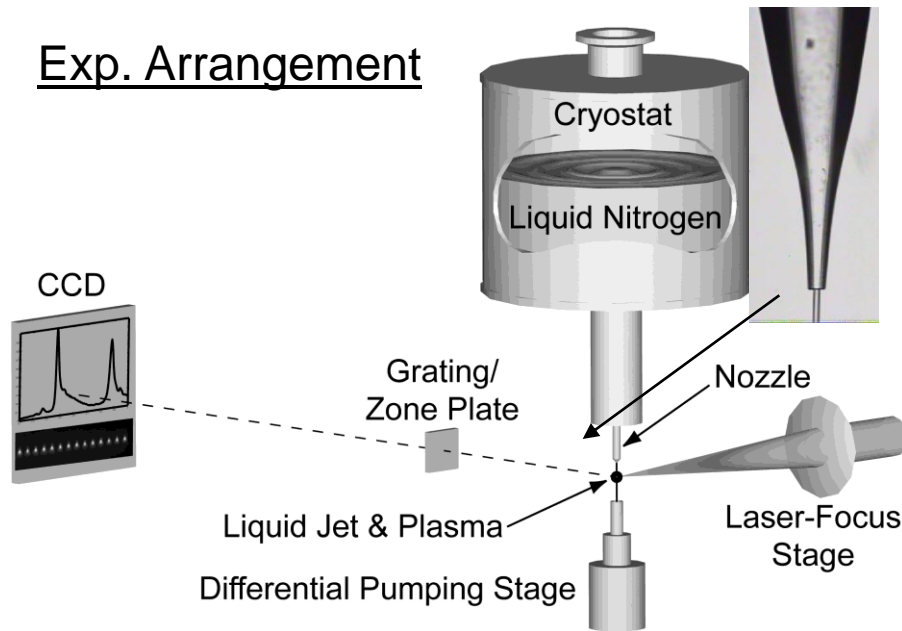
1 h gave coating  
Mitigation need:  $\sim 10^8$

Jansson et al . Appl. Phys. Lett. (2004)

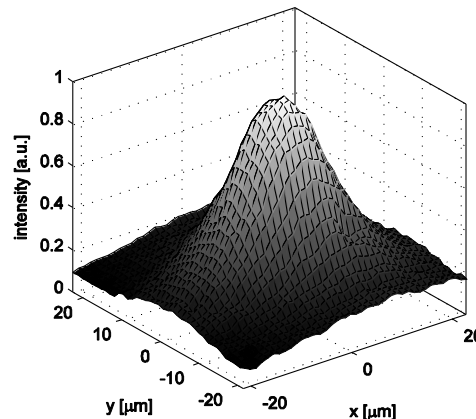
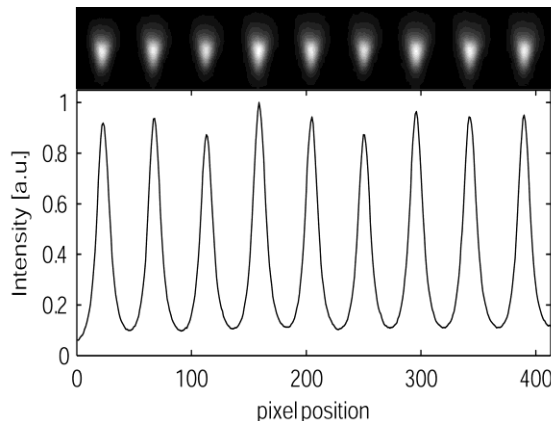


# Water window sources: The liquid-nitrogen-jet source

## Exp. Arrangement



## Stability & Size



Laser: 20W, 100 Hz, 3ns

Flux:  $1 \times 10^{12}$  ph/pulse $\times$ sr $\times$ line

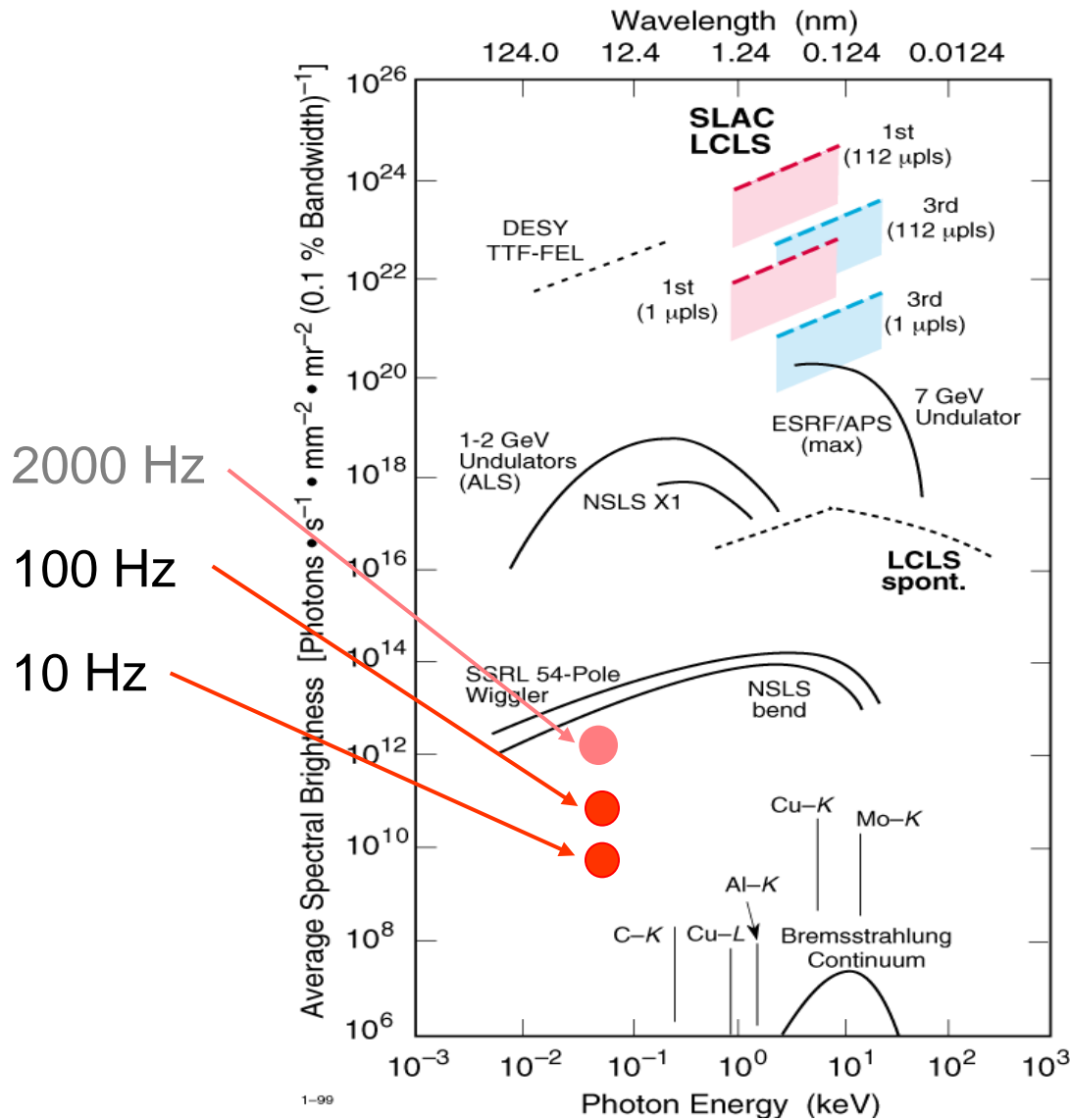
Stability:  $\pm 2$   $\mu$ m

Brightness:  $4 \times 10^8$  photons/  
(pulse $\times$ sr $\times$  $\mu$ m<sup>2</sup> $\times$ line)

Illumination Uniformity: 10%

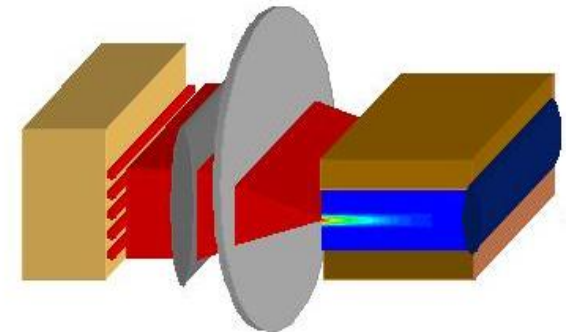
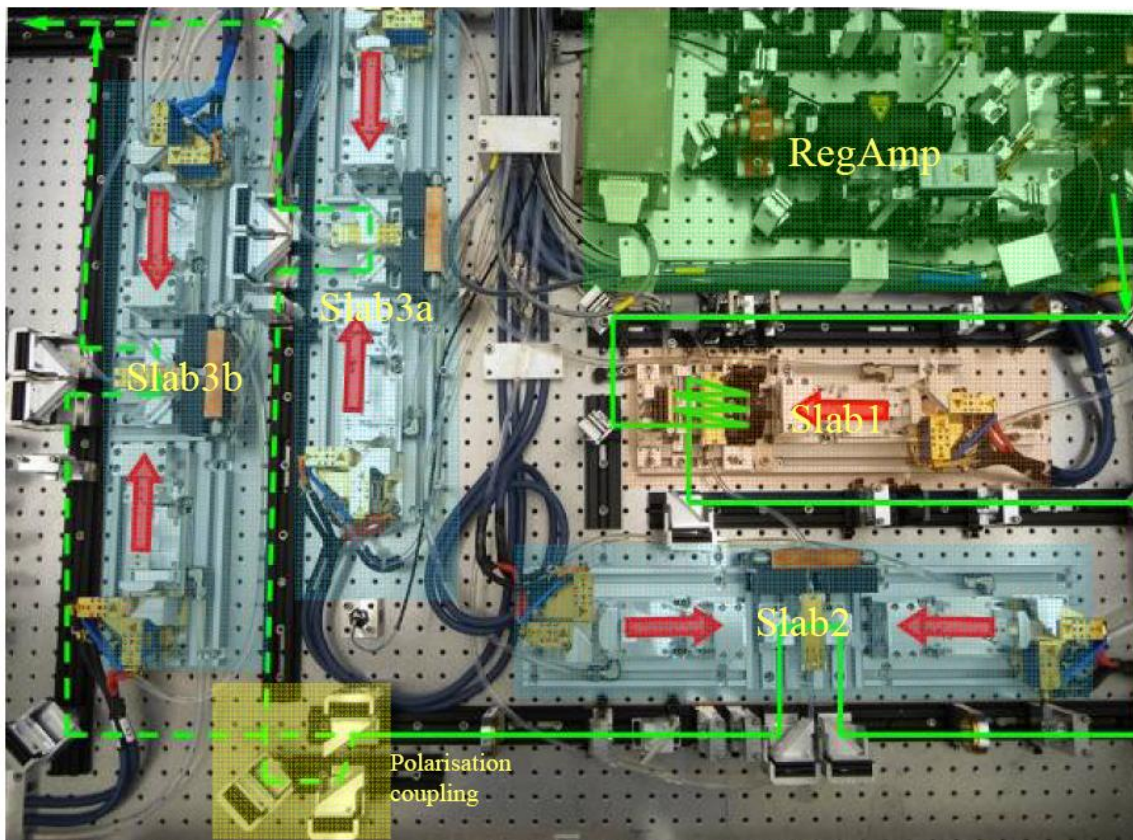
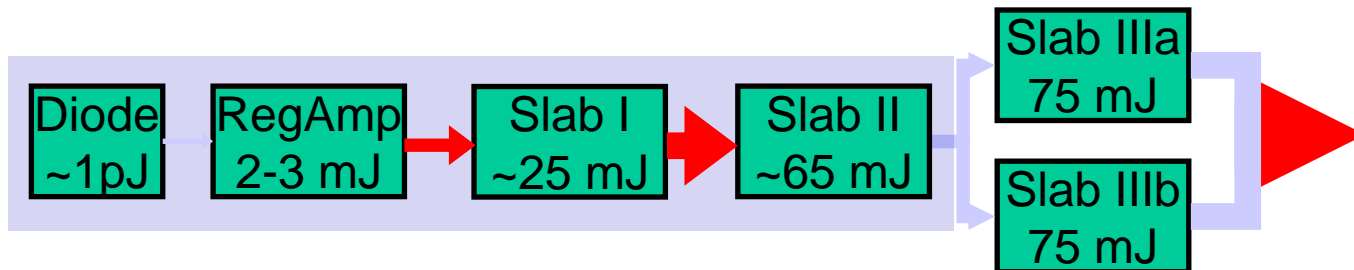


# Liquid-jet laser-plasma sources: Brightness



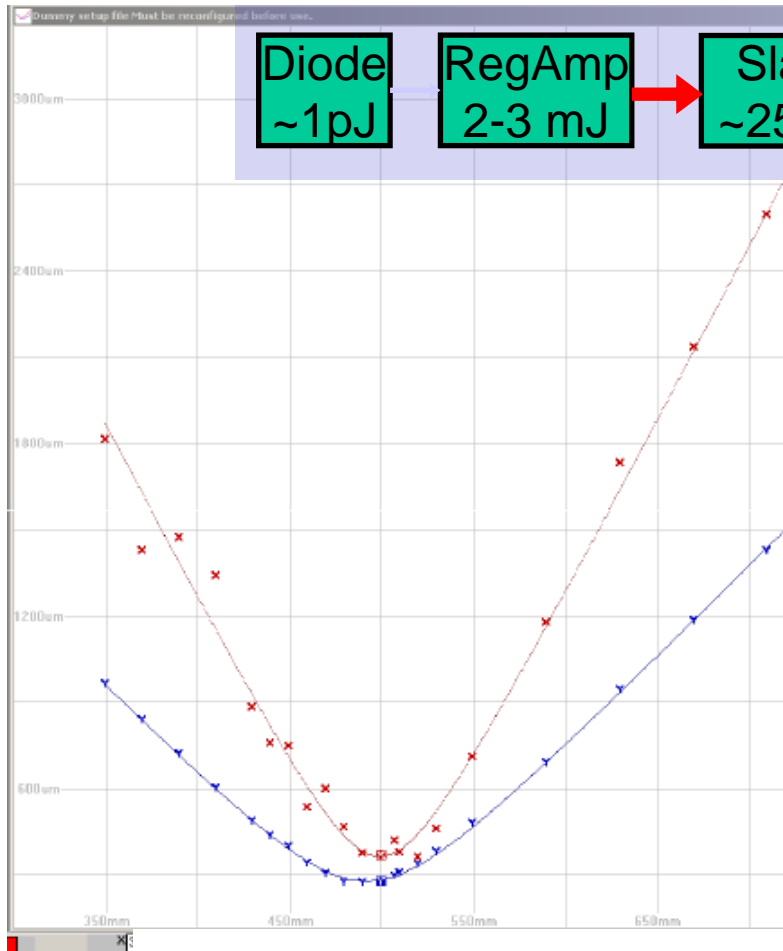
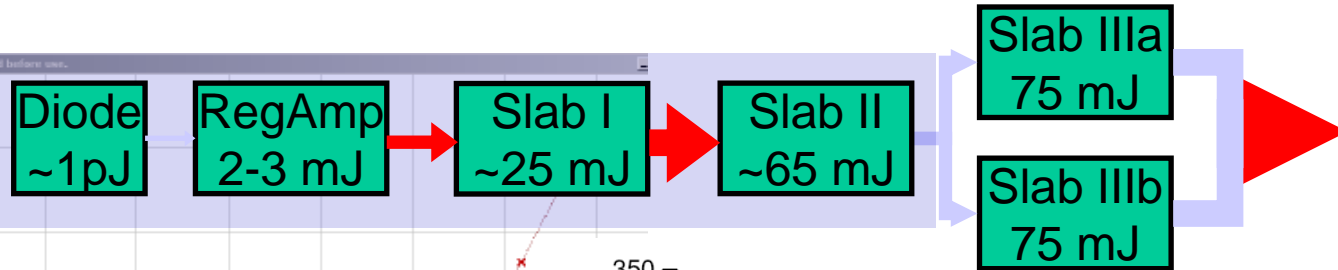
Bright,  
but not like a synchrotron

# Next-generation liquid-jet laser plasmas: 320 W, ~600 ps, 2 KHz Nd:YAG Slab

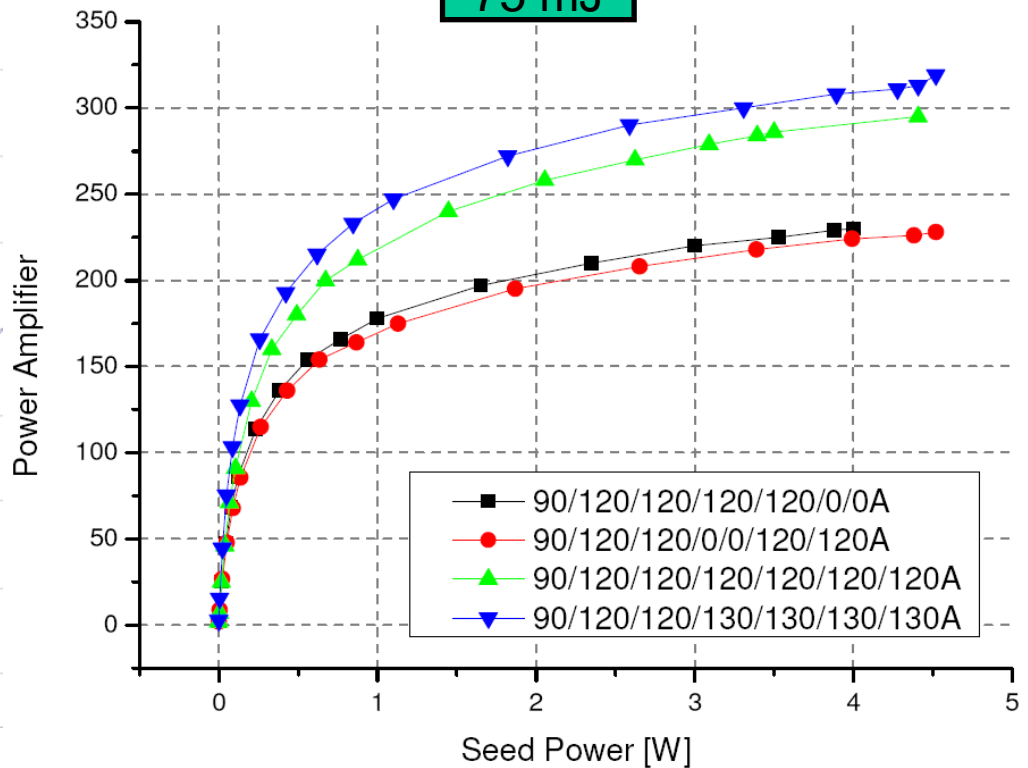


Thanks to D. Esser, M Hofer et al, FhG ILT, Aachen

# Laser Performance



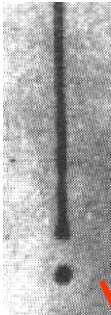
$M^2_{\text{Fast}} = \sim 1.3$     $M^2_{\text{Slow}} = \sim 3.3$



Source scaling to higher power:

# Jet Stability

Laminar



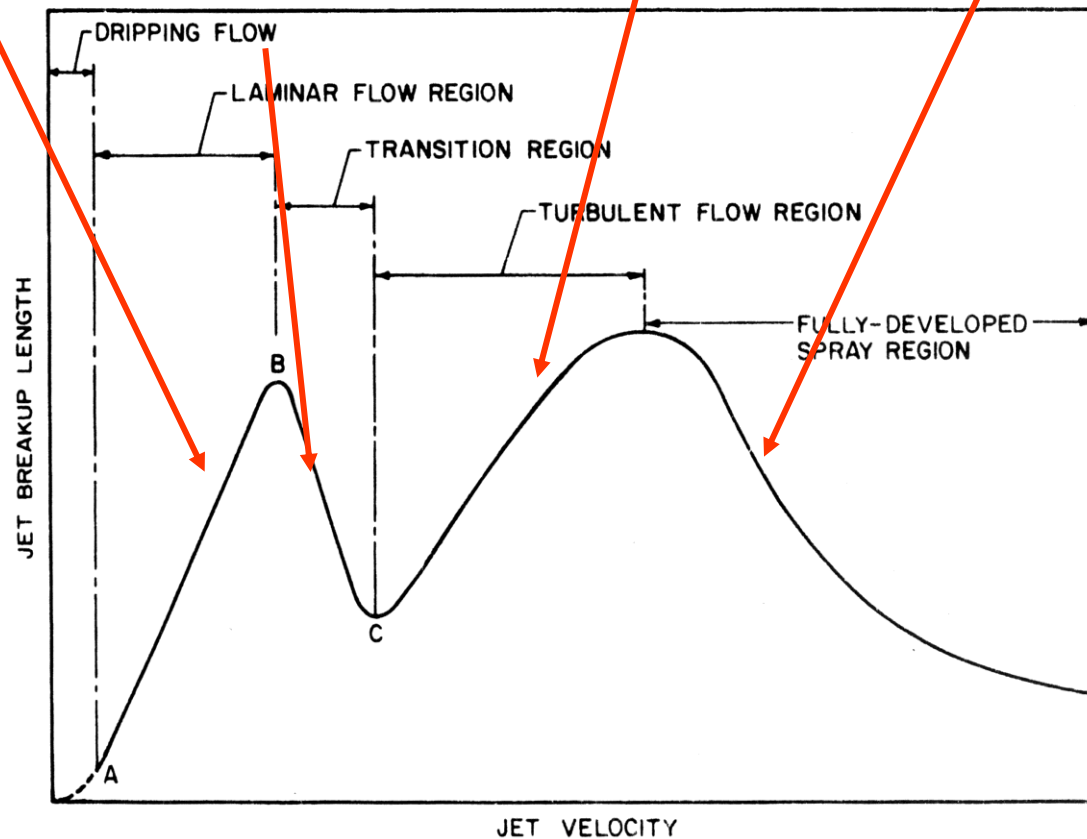
Transition



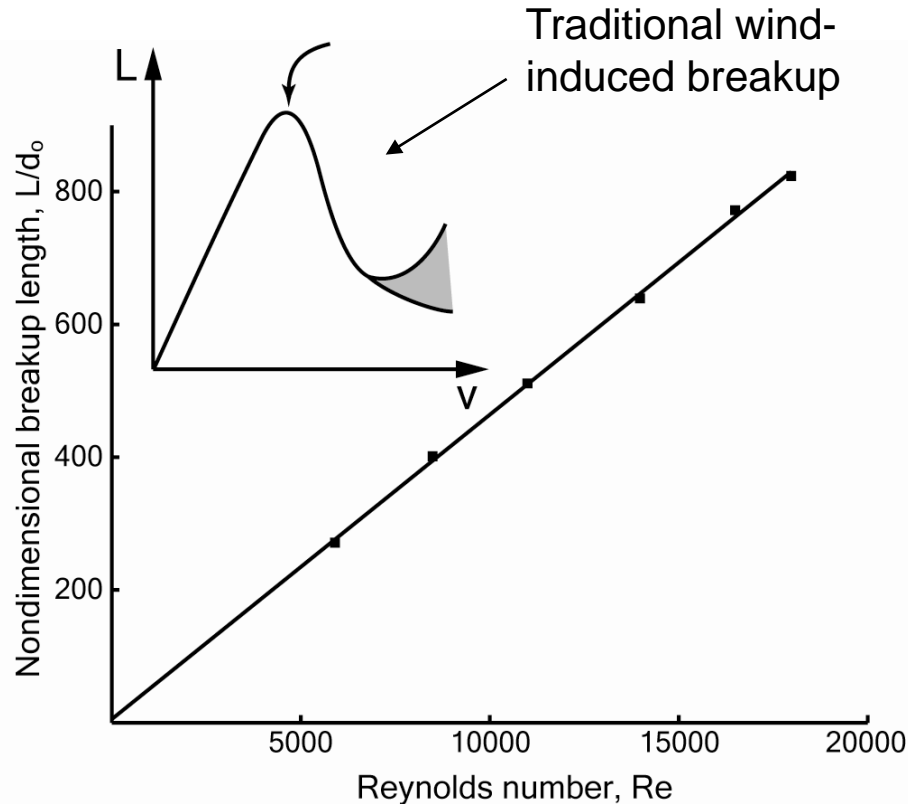
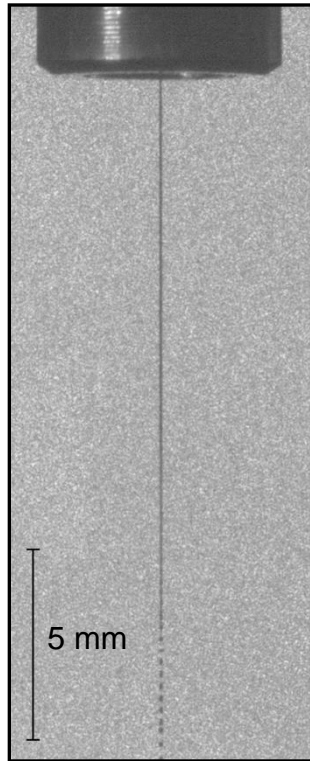
Turbulent



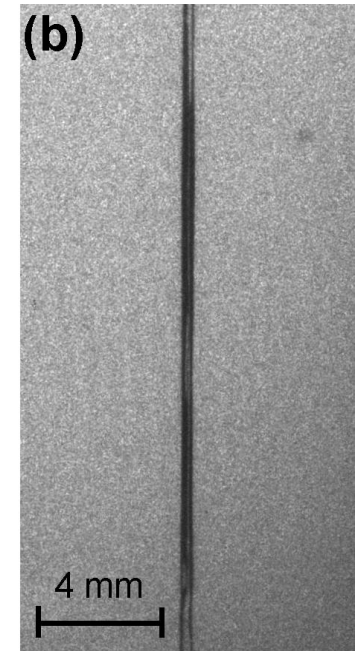
Spray



# Source scaling: Higher jet speed



Stable operation of tin jet to  $Re=18000$   
(diam= $75 \mu\text{m}$ ,  $v=60 \text{ m/s}$ )

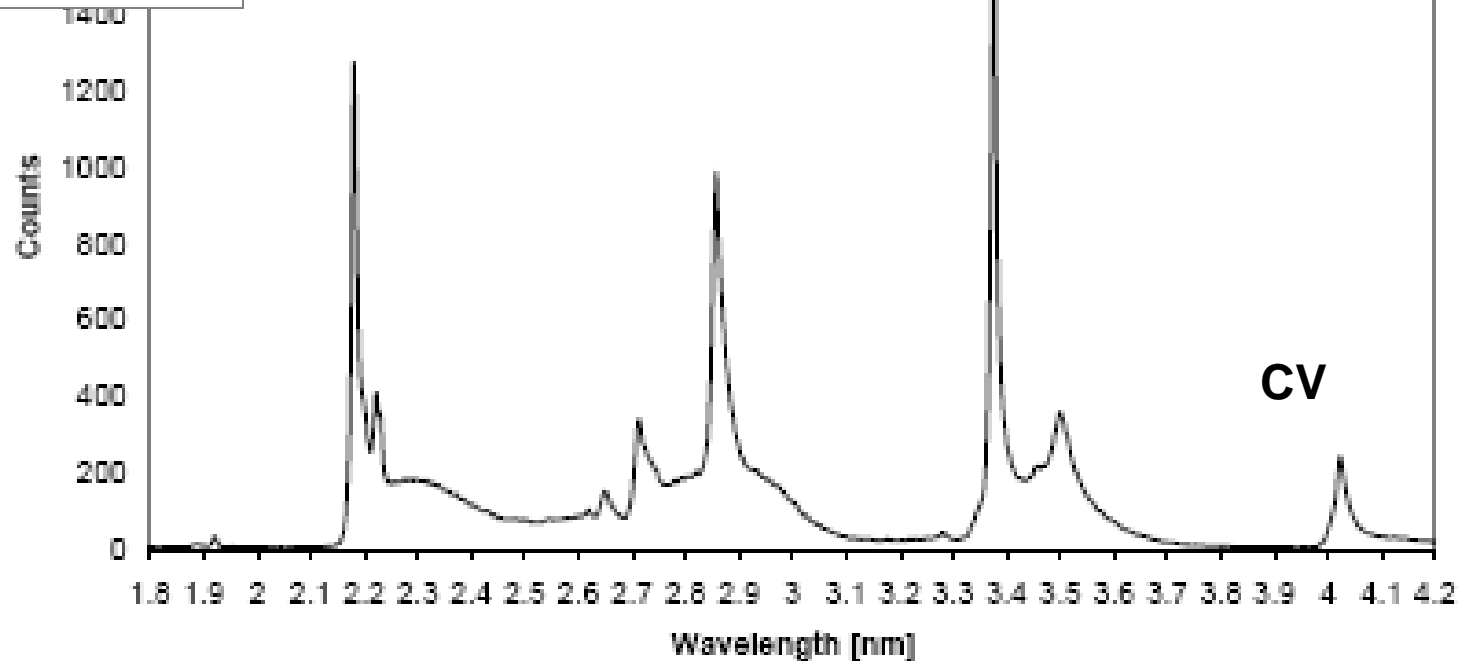
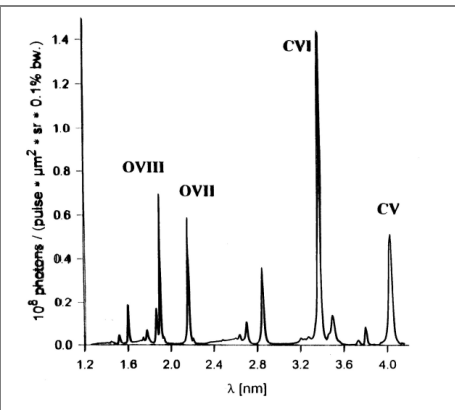


Dynamic similarity experiments with water show that a  $30 \mu\text{m}$ ,  $500 \text{ m/s}$  ( $Re=56000$ ) liquid-tin jet operates stably in vacuum

Otendal et al, Exp. Fluids (2005)

# First methanol-jet spectrum

220 W, 0.8 ns, 2 kHz



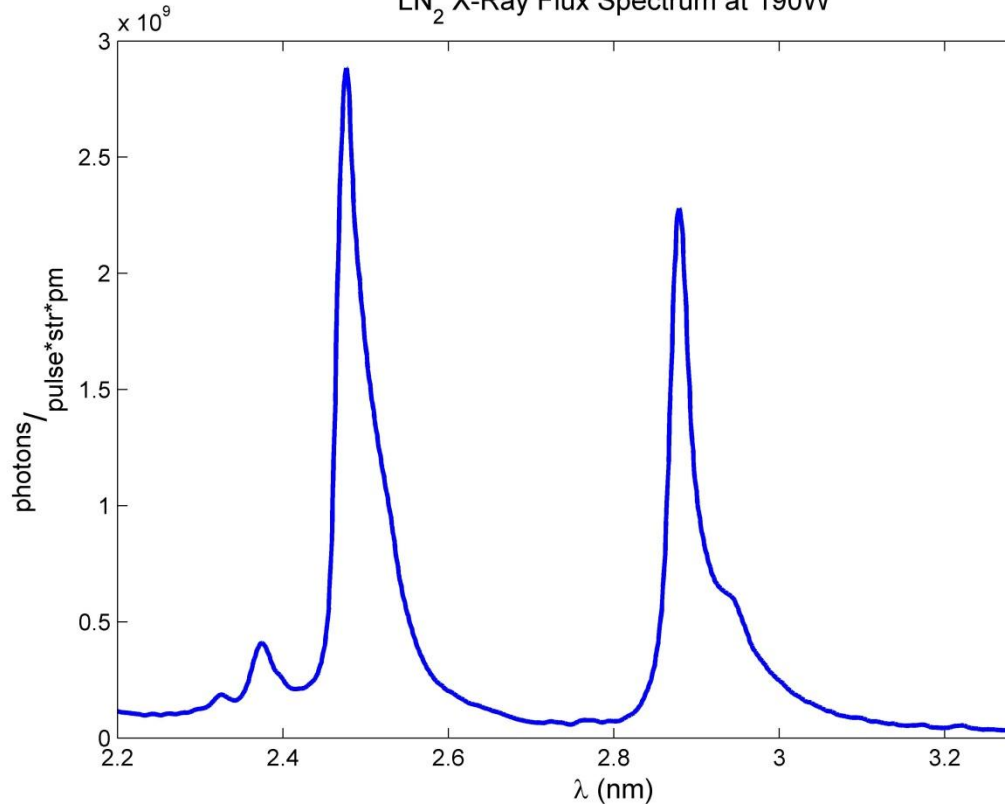
Jet is stable  
Hot plasma

Martz et al, in progress,



# LN<sub>2</sub> Bright Spectrum

LN<sub>2</sub> X-Ray Flux Spectrum at 190W

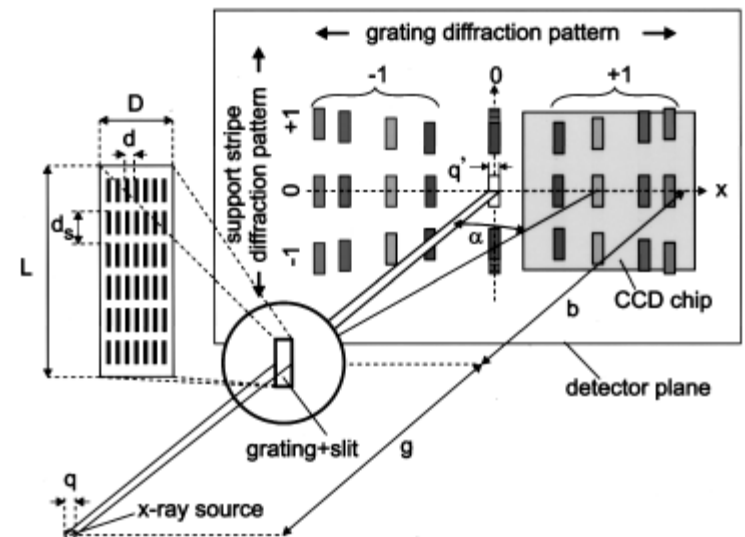


Martz et al, in progress,

CCD & Grating detection calibrated with  
the Max-Born Institute @ BESSY

Geometric Resolution Limit  
 $\Delta\lambda = 0.022$  nm

$\lambda/\Delta\lambda$  at least 700\*



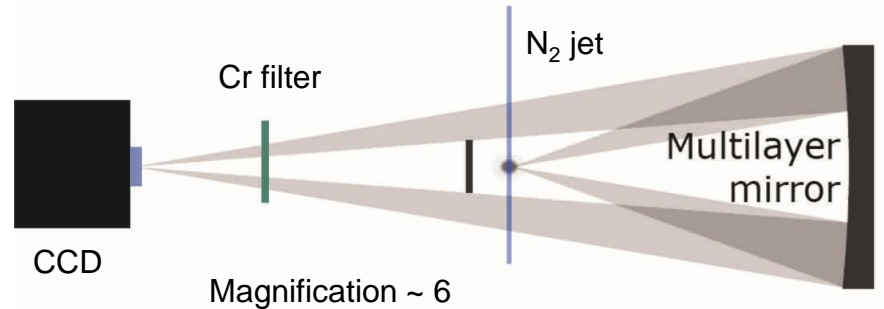
\*T. Wilhein et al, Appl. Phys. Lett. **71**, 190 (1997)

T. Wilhein et al, Rev. Sci. Instrum. **70**, 1694 (1999)

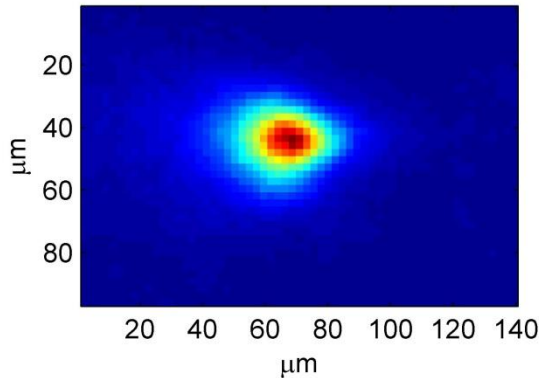


# Laser Plasma Brightness

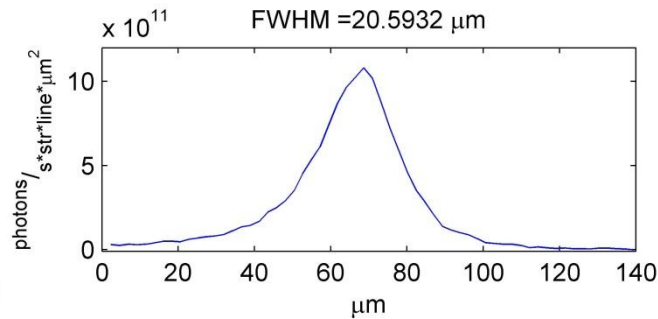
$$\underline{0.5 - 1.5 \times 10^{12}} \frac{\text{photons}}{\text{sec} \times \text{str} \times \text{line} \times \mu\text{m}^2}$$



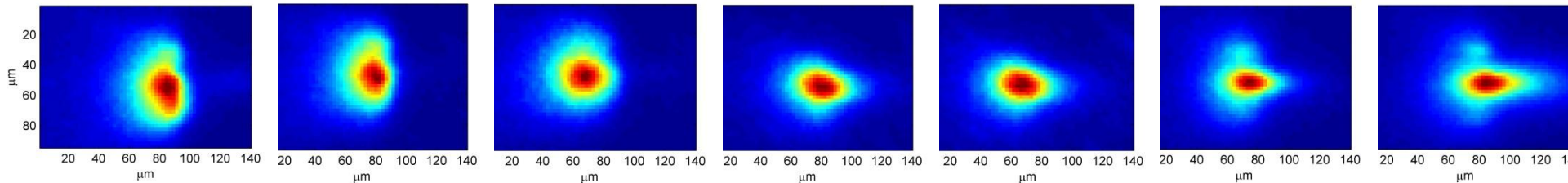
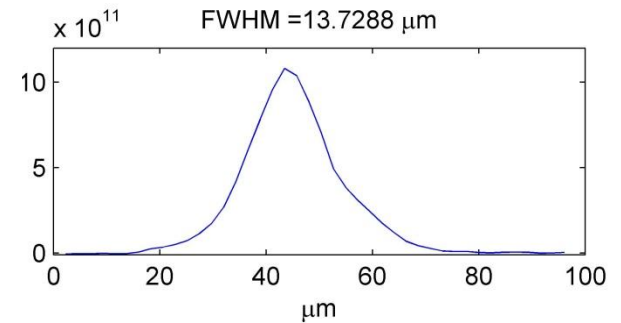
1 Sec Exposure



X - Cut

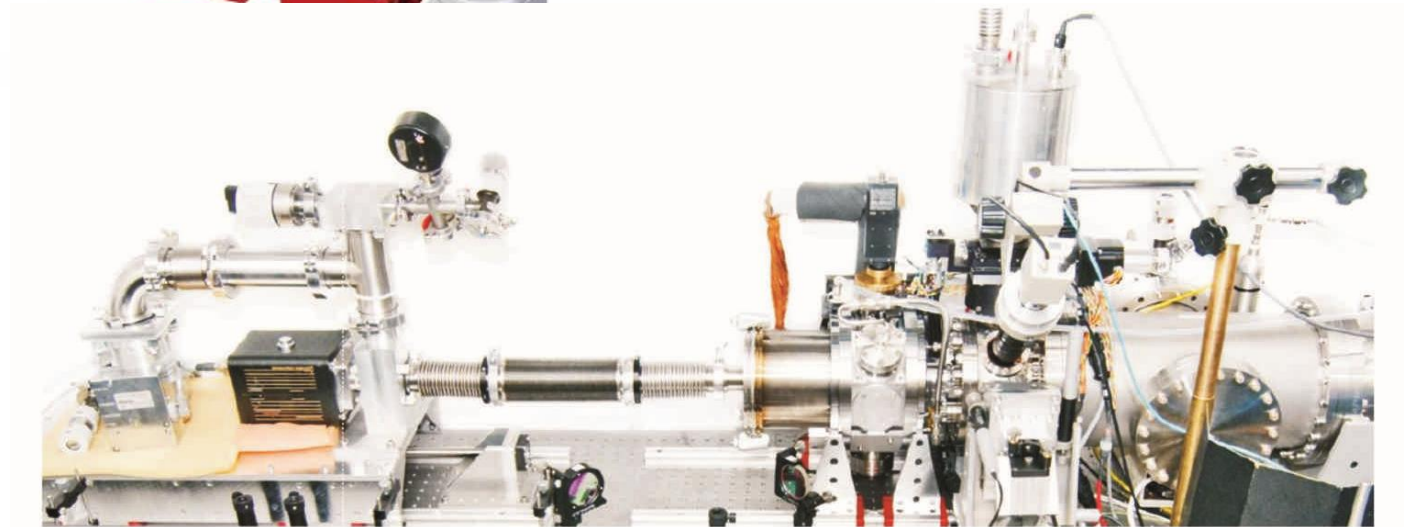
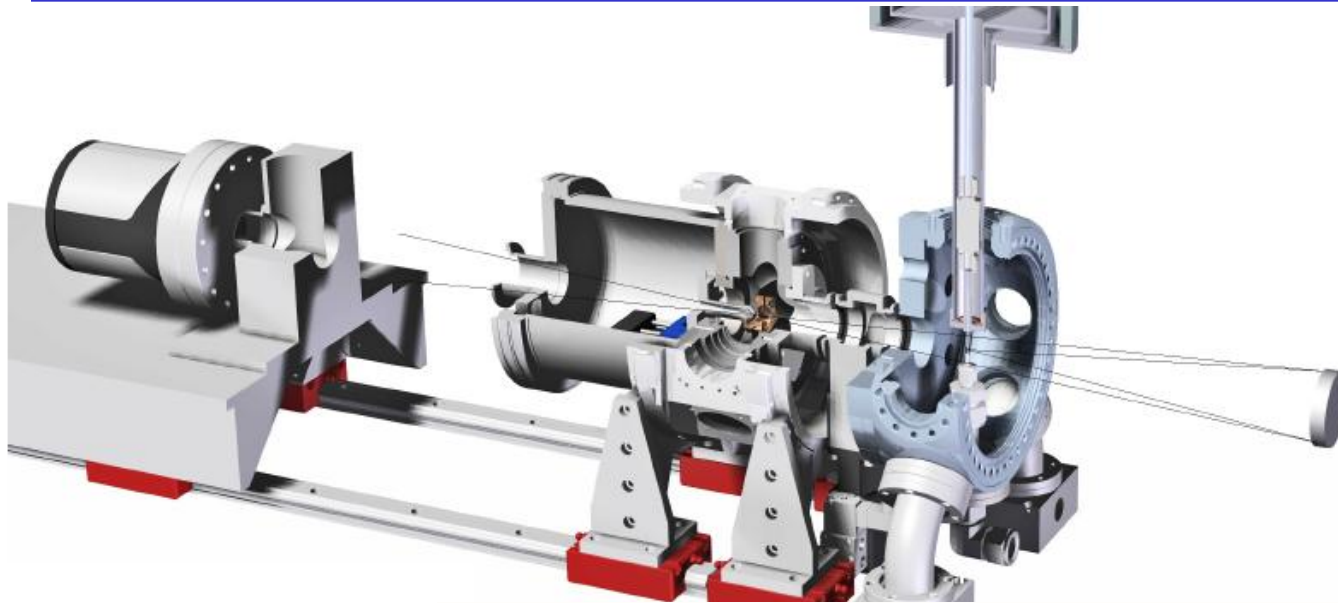


Y - Cut



Martz et al, in progress,

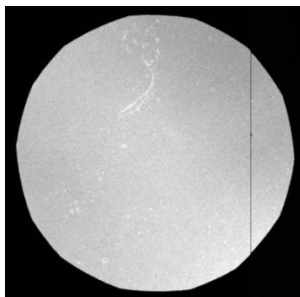
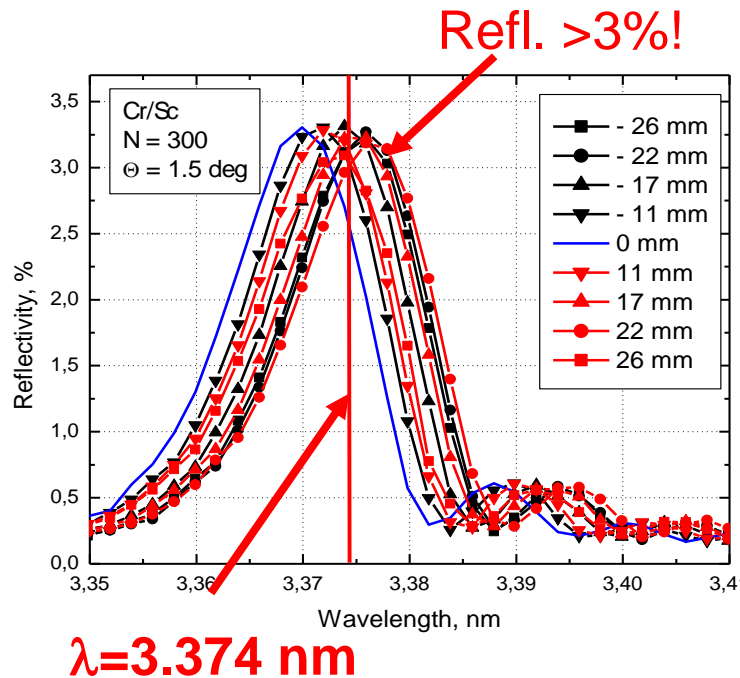
# Soft X-Ray Microscope



2 m

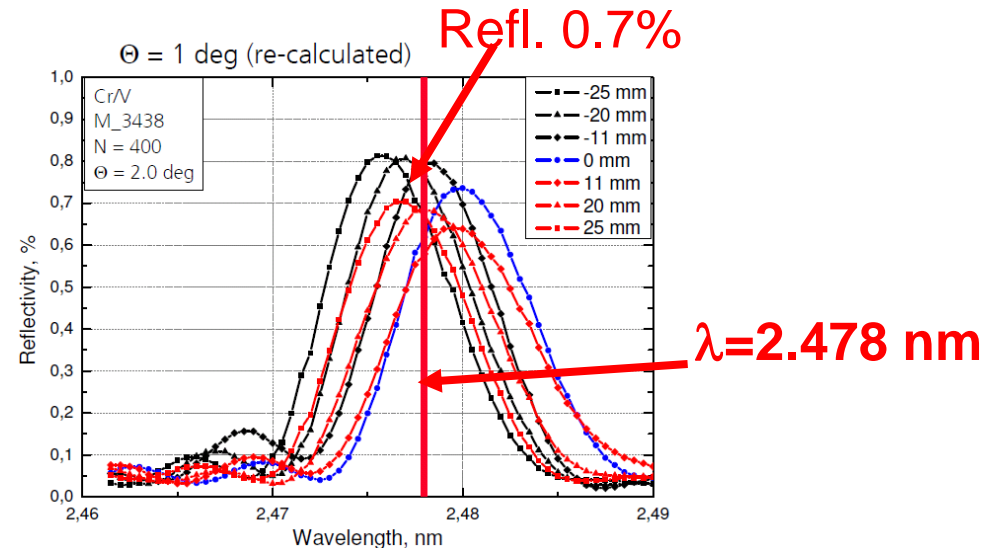
# Normal-incidence multilayer condensers

## Uniformity: Cr/Sc @ 3.374 nm

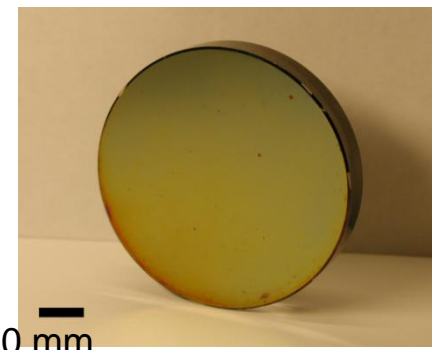


Stollberg et al, RSI (2007)

## Uniformity: Cr/V @ 2.48 nm



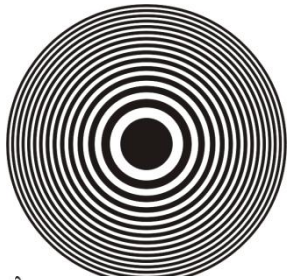
- Cr/Sc
- 58 mm diam  
(active from 20mm - 58mm)
- 350 mm radius
- 200 bilayers
- $\lambda/\Delta\lambda \approx 300$
- Good uniformity
- Good  $\lambda$  match



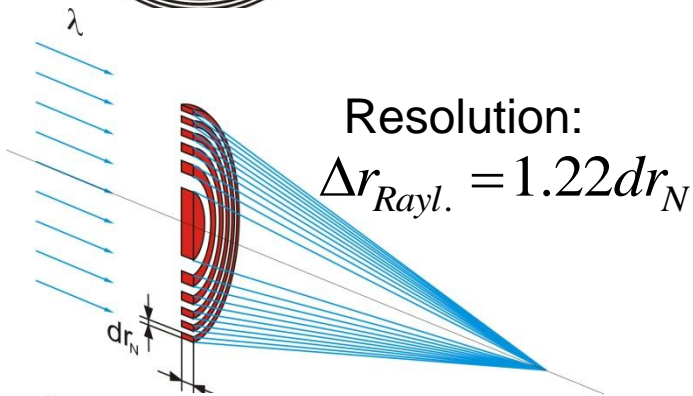
Thanks to S. Yulin, FhG Jena

# Soft x-ray optics: Zone plates

## Basics

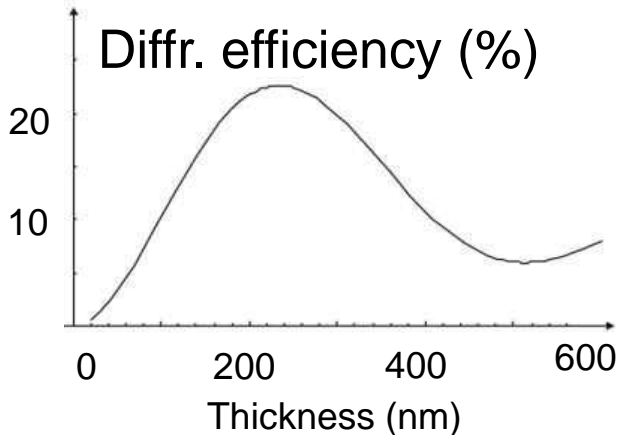


Circular  
diffraction  
gratings

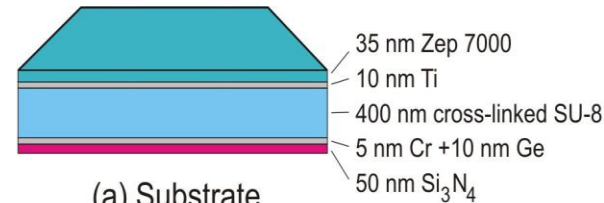


Resolution:  
 $\Delta r_{Rayl.} = 1.22 dr_N$

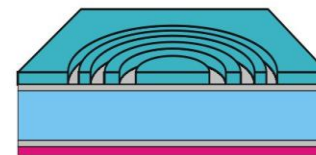
Diffr. efficiency (%)



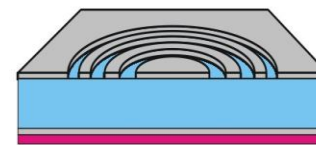
## Nano-fab process for Ni zone plates @ Albanova Nanofab Lab



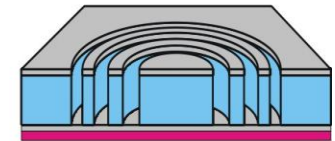
(a) Substrate  
Preparation



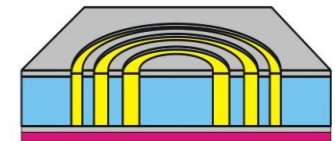
(b) Exposure and  
Development



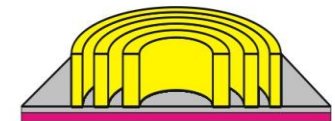
(c) RIE with  $\text{BCl}_3$



(d) RIE with  $\text{O}_2$



(e) Nickel Plating



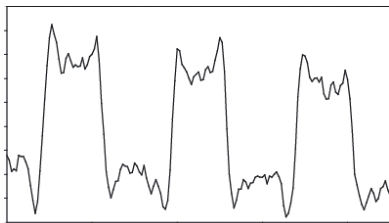
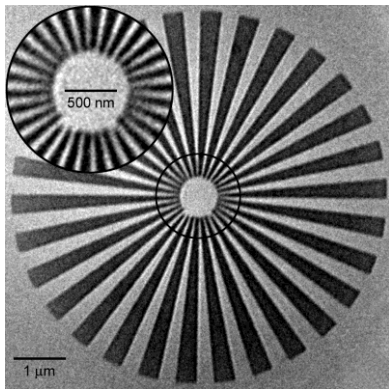
(f) Stripping, RIE  
with  $\text{BCl}_3 / \text{O}_2$

Holmberg et al, MNE (2003),

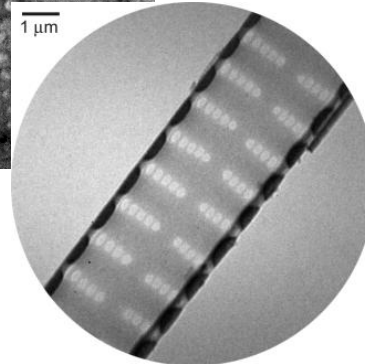
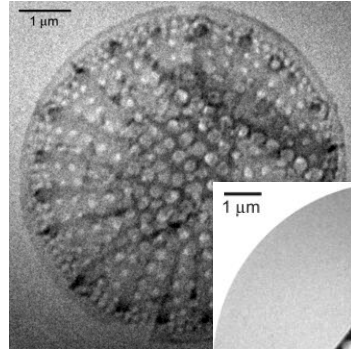


# Laboratory water-window x-ray microscopy

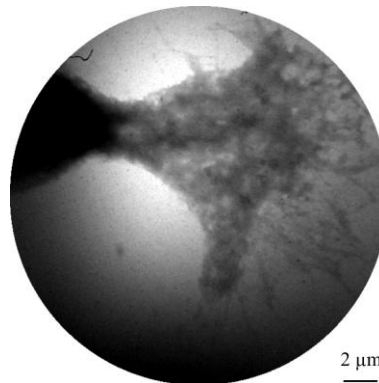
## Test patterns:



## Diatoms:



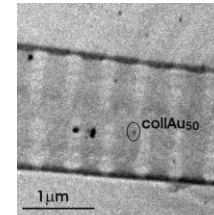
## COS-7 cells



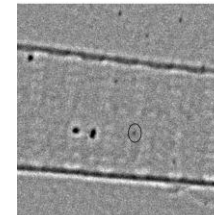
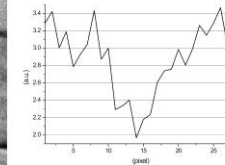
## Function:

Size-selective coll.

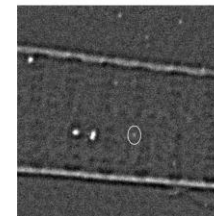
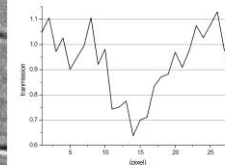
Au identification



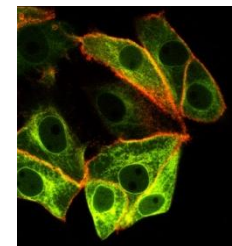
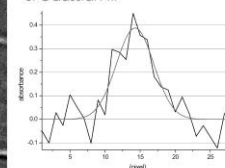
flat-field corrected image



transmission image



absorbance image  
& Gaussian fit



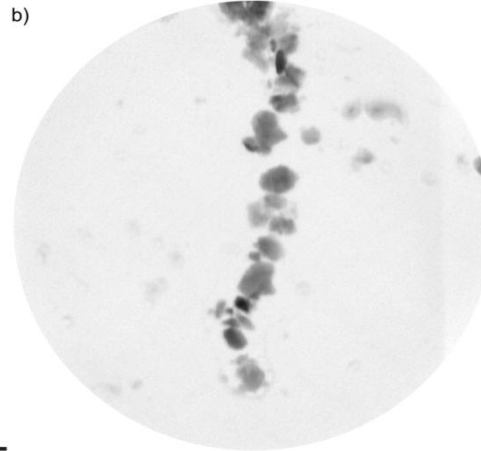
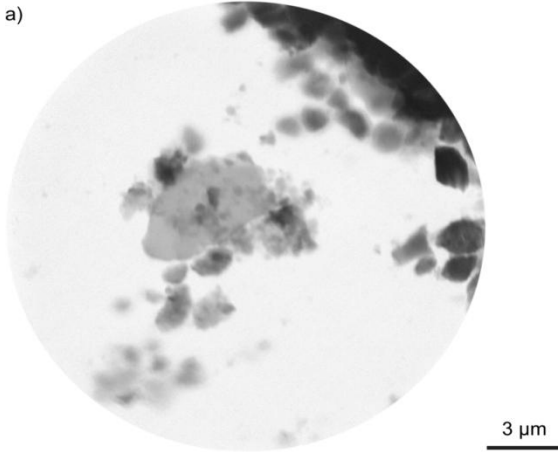
Goal: protein  
co-localization

Recent results:

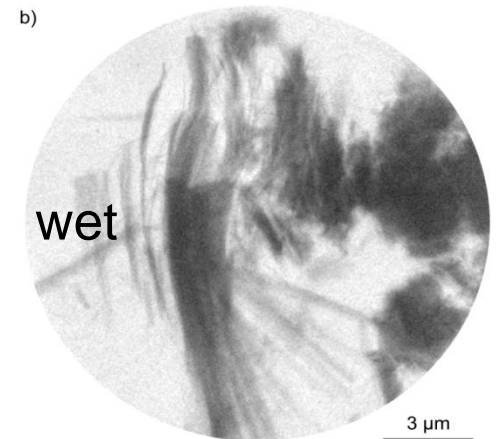
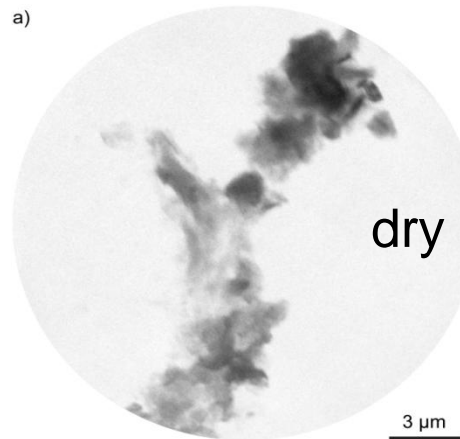
# Environmental colloids

---

## Chernozem, wet



## Nontronite

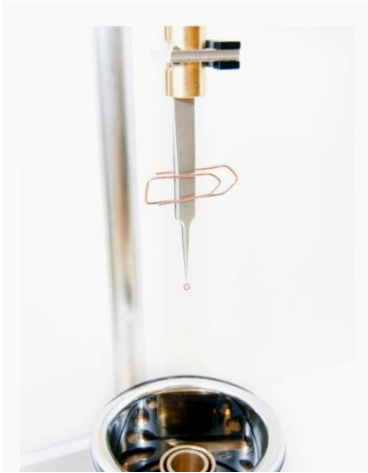


**Thanks to J Thieme et al.!**

Hertz et al, submitted Chem Geol (2010)

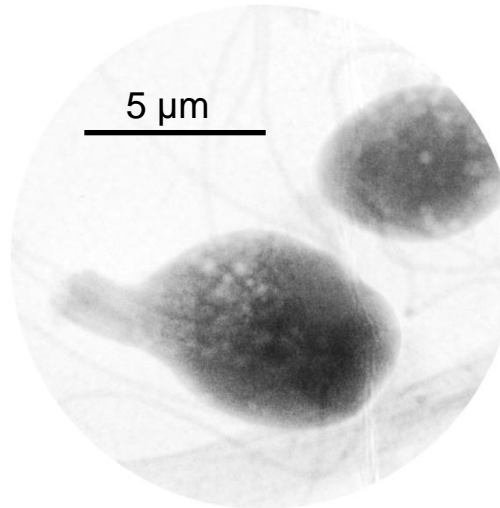
# Recent results: Parasites and cells

## Cryo fixation:

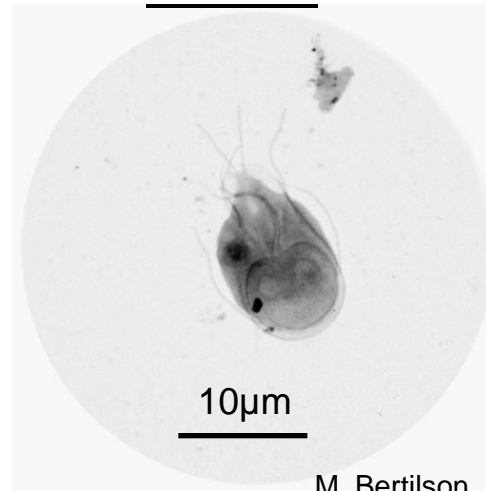


Hydrated samples on TEM grids  
Plunge-freeze in liquid ethane ( $-180\text{ }^{\circ}\text{C}$ )  
TEM high-tilt cryo sample holder

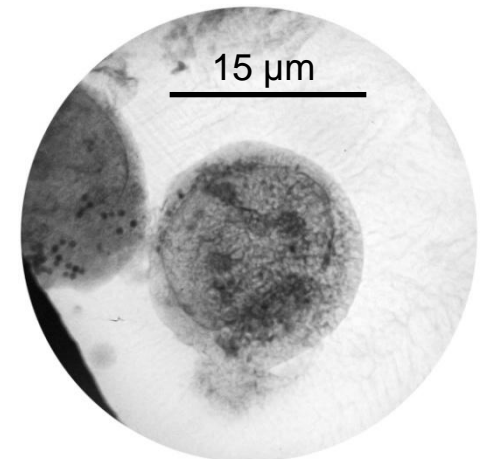
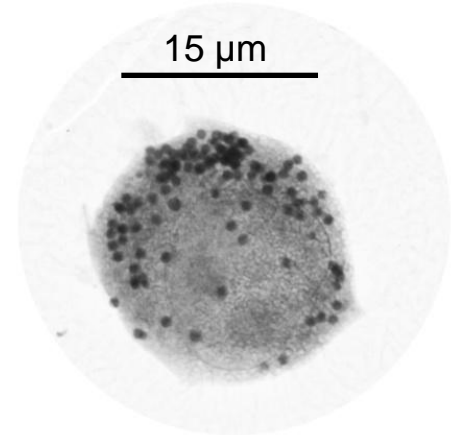
## Spironucleus



## Giardia



## Human immune system B-cells



M. Bertilson, et al, Opt. Lett. Vol. **36**, 2728-2730 (2011)

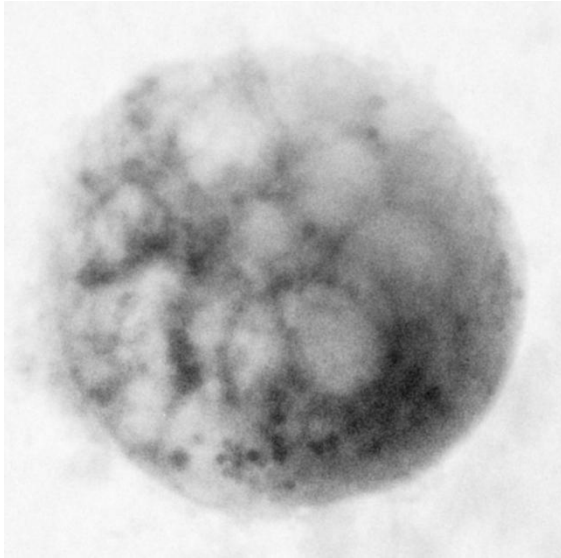


Recent results:

# Laboratory Cryo Tomography: Human kidney cell

---

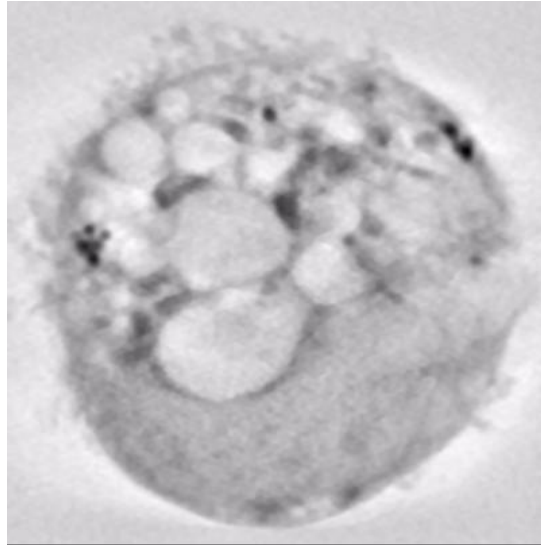
2D image



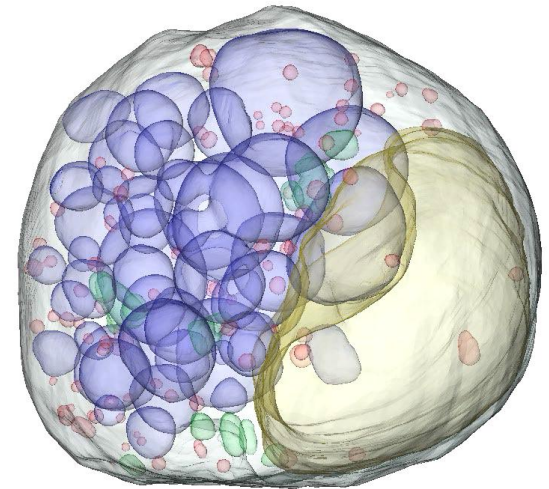
13  $\mu\text{m}$

---

Reconstructed volume



Surface rendering

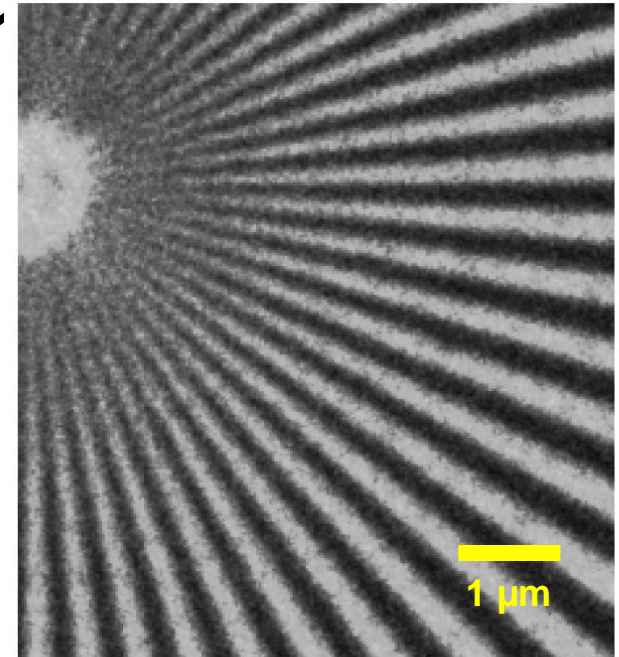
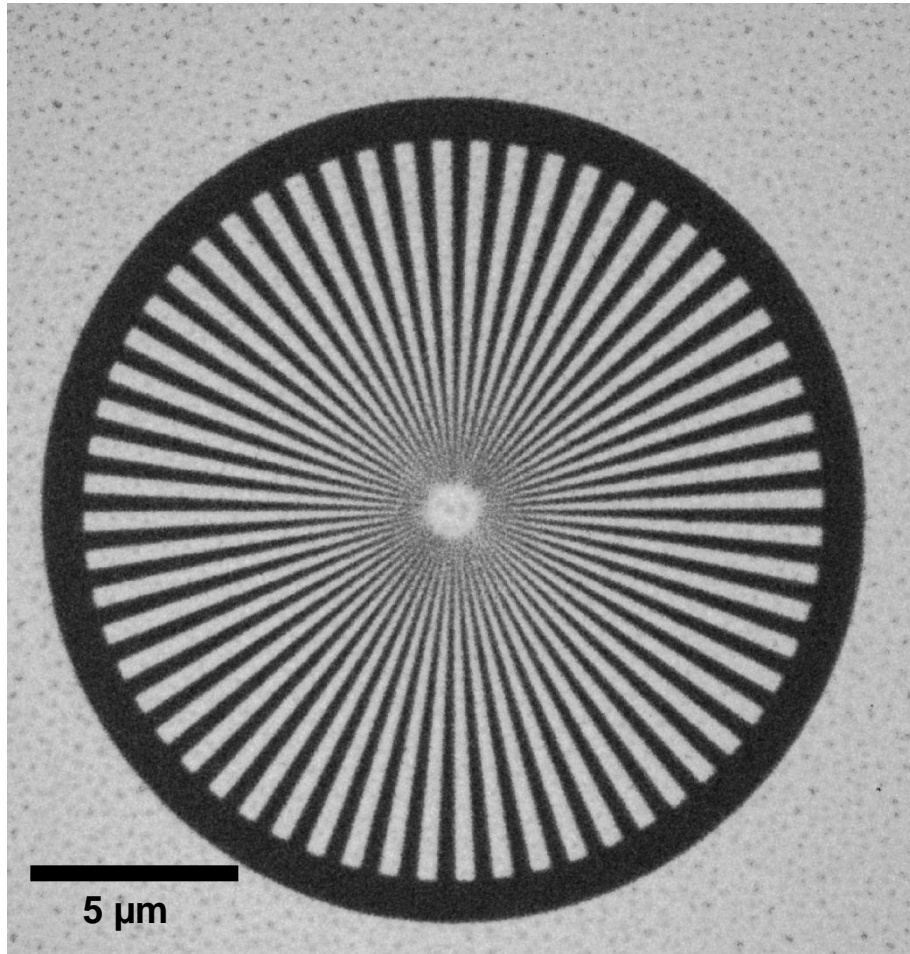


- Tilt series:  $\pm 44^\circ$ , 58 images, 120 s/exp.
- Alignment using landmarks
- Reconstruction with SIRT
- Semi-manual segmentation based on local abs coeff.

M. Bertilson, et al, Opt. Lett. Vol. **36**, 2728-2730 (2011)

# 10 sec Exposure

---



Resolution approaching the  
limit of the ZonePlate  
(~ 40 nm)

# Challenges

---

- Maintaining Focus Quality
  - Stable Laser System is Key
  - Use Appropriate Materials
- Minimize Heating of Jet
- Minimize Etching of Components in Chamber

# Summary

---

## – Laboratory x-ray microscopy approaches synchrotron quality

- Achieved Brightness Comparable to 1<sup>st</sup> Gen Synchrotrons
- Demonstrated preliminary 10 sec exposure times
- Cryo 3D Imaging and Tomography

## – Future Work

- New Zoneplates that Approach 10 nm outer zone width
- Demonstration of more short exposures 10 – 30 sec

## – Always

- Consider full-system imaging properties before choosing optics
- Optimum 3D resolution must be optimised for the studied object

---

# Questions?